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THE HEAT OF FORMATION OF HEXAUREA ALUMINUM III PERCHLORATE

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Picatinny Arsenal Technical Memorandum No. 1295

THE HEAT OF FORMATION OF HEXAUREA ALUMINUM III PERCHLORATE

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ABSTRACT

Hexaurea Aluminum III perchlorate burns smoothly in a combustion bomb leaving an \propto Al₂ 0₃ residue. Calculation of the heat of formation from the reaction:

$$c_6H_{24}o_{18}c_{13}H_{12}Al(s) + 3c_2(g) + 1789.5 H_2O(1) \longrightarrow$$

$$6\infty_2(g) + 6N_2(g) + \frac{1}{2} Al_2O_3(c) + 3 (HCl.600 H_2O)$$

gives a value of -691.53 kcal/mole (at constant pressure and 25°C).

INTRODUCTION

Exploratory combustion experiments with hexaurea Aluminum III perchlorate* indicated that the compound burns smoothly, and leaves a white crystalline residue. An x-ray analysis of this residue showed that only \prec Al₂ O₃ (corundum) was formed**.

Although it was planned to circumvent some of the corrections needed for this type of compound by running comparison experiments it was decided to combust the sample separately because only four 1.2g pellets remained. (A subsequent sample sent to these laboratories caked and turned grey indicating that the aluminum may have separated from the complex).

RESULTS A. Calibration Experiments

The calorimeter was calibrated with Parr Instrument Company bensoic acid (calorific value 6318 cal/g). The bomb was charged with 35cc of As2 03 solution containing the same quantity of HCl as is produced in the sample combustion. A weighed quantity of glass wool saturated with this solution was placed above the sample cup. The bensoic acid was ignited with platinum wire and the rotation was started after the first resistance reading was taken.

Because of these bomb conditions a Washburn correction had to be included for the combustion of bensoic acid. A total of nine corrections were therefore made for each of the five calibration experiments. These corrections are listed in Table 1 and briefly described below.

Step (1) is a correction for the compression of 35cc of solution to 30 atmospheres i.e.



The solution was treated as pure water.

- * This compound was supplied by Allied Chemical, General Chemical Division, Morristown, N.J.
- ** I-ray analysis performed by J. Campisi, Propellant Research Section.

Step 2 - Correction for the compression of bensoic acid i.e.

Step 3 - Correction for the solubility of oxygen in the solution. The solution again being treated as pure $\rm H_2O$ i.e.

Step 4 - Correction for the compression of oxygen i.e.

$$\left[\begin{array}{c} \left(\frac{\partial E}{\partial P}\right)_{T} \end{array}\right]_{0}^{30}$$

Step 5 - Correction for the dilution of the HCl solution with water formed by the reaction.

Step 6 - Correction for the dissolved CO2 in water and its expansion to unit fugacity.

The solution is assumed to be pure water.

Step ? - Correction for dissolved oxygen in water $\triangle E_{soln} O_{2}$ and its expansion to unit fugacity.

Step 8 - Correction for expansion of the gas phase to unit fugacity.

Step 9 - Correction for the decompression of the aqueous phase.

A detailed description of the standardization of calorimeters and the required corrections is described by Neugebauer Ref (1) and by Hubbard, Scott, and Waddington and Prosen Ref (2). The constants used for making these corrections were taken from Ref (2). It should be noted that some corrections are not included because they were deemed insignificant.

B. Combustion Experiments

Combustion of the sample was made in the same environment as in the calibration experiments. Corrections are more complicated than in a routine halogen compound because of the presence of aluminum and nitrogen. 35cc of As2 03 solution was used. A similar quantity of glass wool was saturated with this solution (as in the calibration experiments) and the remaining solution placed in the bottom of the bomb. The corrections which were made are shown in Table 2 and are explained below.

Step 1 - Correction for the vaporization of water before firing.

Step 3 - Correction for the compressibility of the substance was not made. The magnitude of this correction is not significant (in these experiments).

Step 4 - Correction for the solubility of O2 and N2 in the solution (treated as pure H2O). (\(\sigma \subseteq \sigma \

Step 5 - Correction for the compression of the oxygen i.e.

Step 6 - Correction for the solution of ω_2 in the solution (treated as pure water) i.e. $\omega \in \mathbb{Z}$

Step 7 - Correction for the solubility of $\rm O_2$ and $\rm N_2$ in solution (treated as pure $\rm H_2O$).

Step 8 - Correction for decompression of the liquid phase.

$$\left[\left(\frac{\partial E}{\partial P}\right)_{T}\right]_{30}^{\prime}$$

Step 9 - Correction for nitric acid. Aliquots of each of the four bombs washings were mixed and the nitric acid content was analyzed by the Devarda Method References (4).

Step 10 - Correction for the dilution of HCl to 600 \rm{H}_2O_{\bullet}

Step 11 - Correction for the reduction of Cl₂ to HCl via

Step 12 - Correction for decompression of gaseous phase i.e.

If the rotation of the bomb is not started at the mid time of the calorimetric experiment and continued through the end of the final rating period a correction for rotation should be included. In the combustion experiments the rotation was started, (approximately), 63 seconds before the mid time. In the calibration experiments the comparative period is 62 seconds. The estimated heat input from bomb rotation, during this period (62-63 seconds) is less than 2 calories. If the correction is therefore omitted from both the calibration and combustion experiments it will not affect the results significantly.

DISCUSSION OF RESULTS

No attempt will be made to estimate the accuracy of the calculated heat of formation because the recovered chloride in the wash water varies from 92.2% to a maximum of only 96.3% of the calculated value. In one determination where this laboratory found 0.509 g chloride an independent check by the Analytical Section yielded 0.497 g. The Volhard Method was used in both cases. Bubbling the gases of combustion through a NaOH solution showed no chloride indicating that none was lost in the exhaustion process. Washing the Al203 with hot water also showed the absences of chloride. Based on this evidence it must be concluded that the sample purity is questionable and that the result obtained must be used as an approximate value.

It should be noted that in the two determinations where the gases were analyzed for CO2, 98% of the carbon was accounted for. Add to this the solubility of this gas in 35cc of solution remaining in the bomb and one can anticipate a recovery approximating the calculated value.

One additional observation is worthy of note and that is the final form of the Al_2 0_3 . Although x-ray analysis showed only \propto crystals one cannot assume that the amorphous form is completely absent. This assumption seems to be made by Snyder and Seltz in their work on the heat of formation of Al_2 0_3 Reference (3). No attempt was made to quantitatively recover the Al_2 0_3 .

EXPERIMENTAL

Equipment

The heat of combustion was measured in a rotating bomb calorimeter. The bomb was made by the Parr Instrument Company, Moline, Illinois, and is platinum lined. The calorimeter is a submarine type manufactured by the Precision Scientific Company for accomodating a stationary bomb. A new bucket was therefore designed which accepts the rotating bomb and fits into the calorimeter bath.

The design of the bucket and rotating mechanism is based on prints obtained from the U.S. Bureau of Mines at Bartlesville, Oklahoma.

Resistance measurements were made with a platinum resistance thermometer and a G-2 Mueller Bridge each of which was purchased from the Leeds and Northrup Company, Philadelphia, Pa.

The method used is standard and is adequately described in Reference 2.

Sample

The sample was obtained from Allied Chemical Corp in Morristown, N.J. and combusted as received. Their chemical analysis of the sample is as follows:

_	Found	<u>Calculated</u>
α. `	15.5	15.5
Al	3.7 - 3.8	3.935
H	3.99	3.528
C	10.75	10.51
N	***	24.51

A subsequent sample received from Allied Chemical Corporation showed a N content of 24.32 - 24.42%.

REFERENCES

- 1. Heugebauer, C.A., Standard Heats of Formation by Rotating and Stationary Bomb Calorimetry. Thesis Submitted for Ph D (Chemistry) University of Wisconsin 1957.
- 2. a. Rossini, F.D. "Experimental Thermochemistry", Interscience Publishers, Inc., N.Y. Vol I 1956.
 - b. Skinner, H.A. "Ibid, Vol. II 1962.
- 3. Snyder, P.E. and Seltz, H. "The Heat of Formation of Aluminum Oxide", Journal American Chemical Soc. 67,683 (1945).
- 4. Reiman, Neuss, Naiman, "Quantitative Analysis "International Chemical Series, McGraw Hill Book Co., N.Y. 1942.

Calibration Data

Mu. (vac) grams AT (corr) ohms HNO ₂ (corr) cals Std States (1) Corr (2) (4) (5) (6) Total Cals.	.997635 .1581027 -1.48 1.89 .08 .3.93 17.98 -2.16 -17.20 -3.32 -2.16	2 1.039035 .1656443 -1.72 1.89 .08 3.93 17.98 2.25 17.89 3.29 2.25 17.89 2.25 17.89	1.061435 .1683906 -1.48 1.89 .09 3.93 17.98 2.29 18.26 3.27 2.29 2.29 2.29 2.29 2.29 2.29 2.29	972285 .1548271 -1.35 1.89 .08 3.93 17.98 2.10 16.78 3.33 21.56 22.00	1.067680 1.69340 -1.72 1.89 3.93 17.98 2.31 18.58 3.27 21.96	
Water Equivalent Cals/Ohm	610,04	39,783	39,976	39.828	39,849	39,891 £ 45

Sample Data

TABLE 2

Run No.		7	2	3	47	
WT (vac)	grams	1.2256406	1.2093636	1,1955906	1.2161406	
△ T (corr)		.04258226	.04116624	.04068581	.04122991	
CO2 calculated	grams	}	1	.46015	.46807	
CO2 recovered	grams	!	1	.45150	.45730	
Cl calculated	grams	İ	.18750	.18536	.18855	
Cl recovered		!	.17724	.17086	18149	
% CO2 recovered		-	į	98.1	7.76	
% Cl recovered		4	64.5	92.2	96.3	
Standard States	(1)	4.26	7.56	4.26	4.26	
(corr)	(5)	-1.89	-1.89	-1.89	-1.89	
	(3)	!	1	!	1	
Cals	(1)	-3.93	-3.93	-3.93	-3.93	
	(5)	-17.98	-17.98	-17.98	-17.98	
	<u>(9</u>	3.38	3.33	3•30	3.35	
	(2)	70.4	4.07	4.07	70.4	
	(8) (8)	2.05	2.02	2.02	2.02	
	(6) (6)	None found				
	(or)	16	15	14	15	
	(II)	35.09	31.18	27.0%	32.39	
	(12)	20.45	20.53	20.50	20.56	
,	(13)	-4.34	-4.34	-4.34	-4.34	
Total (corr) G	als	16.07	37.10	26.93	38.36	
△T × 39,891		-1698.65	-1642.16	-1623.00		
	cal/g	-1352.55	-1327.19	-1334.96	•	1333.9
Heat liberated	KCAL/MOLE	4.1.26-	-910-0	-415.3	•	414.0

 \triangle H_c (corr to std pressure) kcal/mole =

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